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Research Article

Zn-Mo/HZSM-5 Catalyst for Gasoil Range Hydrocarbon Production by Catalytic Hydrocracking of *Ceiba pentandra* Oil

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Abstract

Biofuel from vegetable oil becomes one of the most suitable and logical alternatives to replace fossil fuel. The research focused on various metal ratio Zinc/Molybdenum/HZSM-5 (Zn-Mo/HZSM-5) catalyst to produce liquid hydrocarbon via catalytic hydrocracking of *Ceiba pentandra* oil. The catalytic hydrocracking process has been applied in this study to crack *Ceiba pentandra* oil into a gasoil range hydrocarbon using Zn-Mo/HZSM-5 as a catalyst. The effect of various reaction temperature on the catalytic hydrocracking of *Ceiba pentandra* oil were studied. The Zn-Mo/HZSM-5 catalyst with metal ratio was prepared by incipient wetness impregnation method. This process used slurry pressure batch reactor with a mechanical stirrer. A series of experiments were carried out in the temperature range from 300-400 °C for 2 h at pressure between 10-15 bar. The conversion and selectivity were estimated. The liquid hydrocarbon product were identified to gasoline, kerosene, and gas oil. The results show that the use of Zn-Mo/HZSM-5 can produce gas oil as the most component in the product. Overall, the highest conversion and selectivity of gas oil range hydrocarbon was obtained when the ZnMo/HZSM-5 metal ratio was Zn(2.86 wt.%) - Mo(5.32 wt.%) / HZSM-5 and the name is Zn-Mo/HZSM-5₁₀₂. The highest conversion was obtained at 63.31 % and n-paraffin (gas oil range) selectivity was obtained at 90.75 % at a temperature of 400 °C. *Ceiba pentandra* oil can be recommended as the source of inedible vegetable oil to produce gasoil as an environmentally friendly transportation fuel. Copyright © 2018 BCREC Group. All rights reserved

Keywords: Catalytic hydrocracking; *Ceiba pentandra* oil; Zinc/Molybdenum/HZSM-5 catalyst; Liquid hydrocarbon

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1. Introduction

Continuous use of fossil fuels creates a negative impact on the environment such as acceler-

ate global warming, CO₂ emissions, greenhouse effects and air pollution and other environmental problems [1-4]. One of the efforts made to overcome the energy crisis is to develop fuel from sources of renewable energy, to develop fuel from new energy sources. One of the most

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promising alternative fuels is biofuels. Biofuels are defined as solid, liquid or gaseous fuels derived from biomass substrates as renewable or flammable feedstock, and may be used as (partial) substitutes for fossil fuels [5]. In Indonesian regulation (KEPRES) 22 /2017, on a primary national energy plan concerning national energy development. Biofuel is considered as an alternative and clean fuel to replace petroleum [6,7]. There are several studies inedible oil as promising feedstocks for biofuel production. Biofeeds, such as vegetable oil, lignocellulosic biomass, algae, and sewage sludge, can be feedstock to produce biofuel [8]. Inedible vegetable oil as biofeed can be best choice to solve the issue of competition between the use of vegetable oil as the source of food and that of the fuel.

Therefore, some of inedible oil have been investigated and reported [6-11]. One type of vegetable oil that can be utilized to synthesize bio-

hydrocarbons is *Ceiba pentandra* seed oil. *Ceiba pentandra* plant is a tropical plant, easily obtained and cultivated in Indonesia. The *Ceiba pentandra* seed contains about 20-28 % by weight of oil. *Ceiba pentandra* plants can produce up to 1160 kg of seed per hectare per year assuming 25 % oil in seeds. The productivity of *kapok* oil is approximately 70 % of the soybean productivity oil. So far, *kapok* seeds has become untapped waste maximally. *Ceiba pentandra* seed oil is not used as food, because it has a chemical nature that is easy to change [11]. *Ceiba pentandra* oil is extracted from *kapok* seed, as shown in Figure 2(b) and (c). *Kapok* plant is also called the Java *kapok* plants, *kapok* Java, or silk *kapok* trees. Characteristics of *Ceiba pentandra* tree crops among others have a high up to 70 meters. The *Ceiba pentandra* plant besides producing fiber also has the potential to produce oil. The *kapok* seeds are

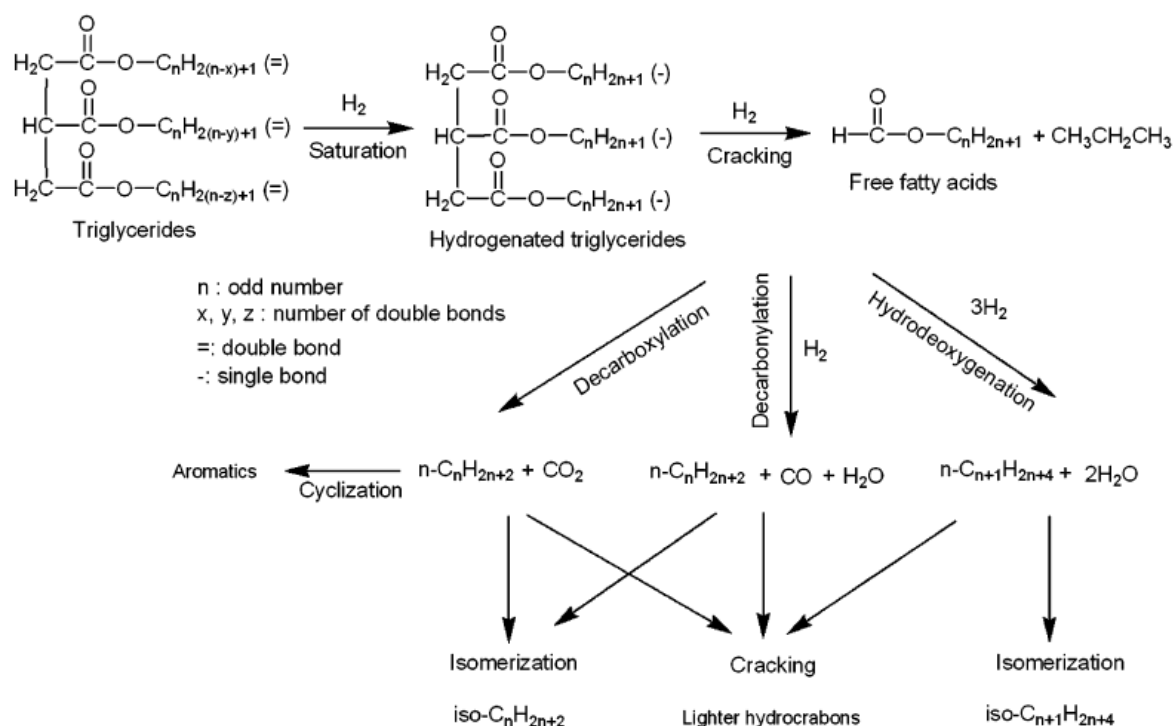


Figure 1. Triglyceride mechanism using hydrotreating catalyst [14]

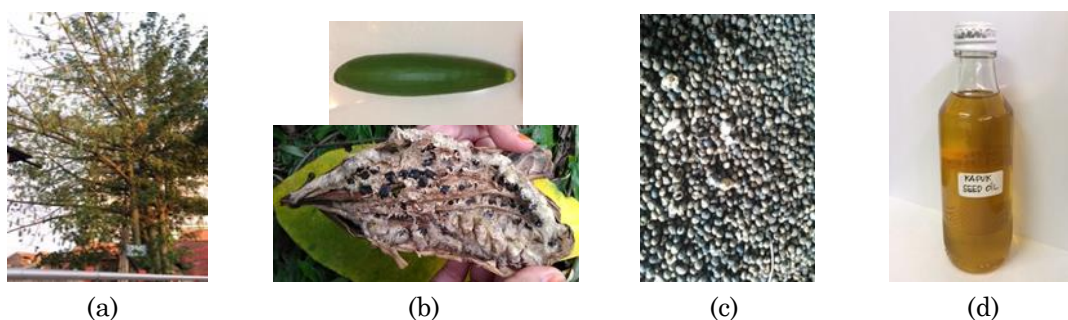


Figure 2. *Ceiba pentandra* (a) plant, (b) fruit, (c) seeds, and (d) oil

blackish, consisting of water content of 13 %, 5 % ash, 20 % crude fiber, 6 % fat, 29 % protein, and 20 % carbohydrates with clean texture and not clot.

The catalytic hydrocracking process is one of the alternative processes for producing biofuels, because it can convert vegetable oils into hydrocarbons in particular C₁₅-C₁₈ using bi-functional heterogeneous catalysts. Catalytic hydrocracking removes double bonds and carboxyl groups in fatty acids to obtain n-paraffin compounds [12]. The process method of catalytic hydrocracking is the incorporation of catalytic cracking process with the addition of H₂ gas. The method is capable of producing biofuels at high temperatures (300-400 °C) and requires more energy for the process of cracking vegetable oils into hydrocarbons, but producing products that have high oxidation stability and cetane number [10]. The catalytic hydrocracking process involves a hydrodeoxygenation reaction as the main reaction and decarboxylation as a side reaction [13]. Liquid hydrocarbon products generally contain a n-alkane-rich chain of C₁₅-C₁₈ straight through three different reaction stages: decarbonylation, decarboxylation and hydrodeoxygenation [14]. The conversion process of vegetable oils through the catalytic hydrocracking method involves catalysts to speed up the reaction rate and increase selectivity. The conventional catalysts commonly used in the catalytic hydrocracking process are alumina supported by sulphide forms, such as NiMo or CoMo [14-17]. The alumina catalyst (Al₂O₃) is amorphous and has a texture of a smaller surface area and has a lower acid site when compared with zeolite. Zeolite catalysts have hydrophobic properties that serve to assist the cracking process of vegetable oil triglycerides [18]. The zeolite catalyst HZSM-5 has a strong acid site at high temperatures, capable of forming paraffins and aiding the cracking process [19].

Liquid hydrocarbon biofuel products can be produced from hydroprocessing of carinata oil with Mo-Zn/Al₂O₃ catalyst [17]. Sousa *et al.* [20] also reported that biofuel in diesel range can be obtained from hydrotreatment of sunflower oil using supported molybdenum carbide. However, in the study of Zhao *et al.* still use Al₂O₃ has smaller surface area and the acid site is lower than zeolite. Therefore, to increase the catalyst activity requires a larger surface area so that HZSM-5 was used to improve the catalyst structure in the previous study [17].

In the present work to focus on various metal ratio Zn-Mo/HZSM-5 catalyst via *Ceiba pentandra* oil catalytic hydrocracking for liquid hy-

drocarbon production. It can be said that the product orientation generated in this research leads to a liquid hydrocarbon product with a composition equal to that of petroleum diesel. The product is called gas oil. The effect of Zinc/Mo ratio and reaction temperature process on the conversion and the selectivity of gasoil range hydrocarbon were investigated under a relatively low-pressure condition at 10-15 bar.

2. Materials and Methods

2.1 Materials and chemicals

Ceiba pentandra oil contains unsaturated fatty acids in the form of linoleic acid (C18:2) and palmitic acid (C16:0) were 78.85 % and 21.12 % as a majority composition [21]. *Ceiba pentandra* oil was analyzed using gas chromatography-mass spectrometry with chromatogram as in the previous study [22].

The ammonium-ZSM-5 zeolite were taken from Zeolyst International (CBV 8014, a surface area of 425 m²/g, Na₂O content less than 0.05 wt.%), while Zn(NO₃)₂·6H₂O and (NH₄)₆Mo₇O₂₄·4H₂O was purchased from Merck with ≥98 % purity. The Zn-Mo/HZSM-5 catalyst was prepared with procedure as reported by Mirzayanti *et al.* [21-23].

2.2 Preparation of catalyst

The H-type ZSM-5 was obtained by simply calcining the as-prepared sample at 550 °C for 5 h and named as HZSM-5 as reported by the previous study [21-23]. The sample of HZSM-5 was modified by designed concentration of Zn(NO₃)₂·6H₂O and (NH₄)₆Mo₇O₂₄·4H₂O aqueous solution with *incipient wetness impregnation* method [24]. Measurements of Zinc and Mo metals to HZSM-5 were as follows: HZSM-5 was weighed 4.75 g then dried in oven for 12 h at 120 °C. Zinc nitrate precursor solution was prepared and then added slowly into the HZSM-5 while stirring using a magnetic stirrer at 30 °C for 3 h. The sample was kept so wet that the precursor solution was completely mixed. The catalyst sample was dried in the oven for 12 h at 120 °C. The sample is named as ZincHz. Thus, Mo was loaded on the ZincHz catalyst by impregnation while stirring using a magnetic stirrer at 30 °C for 3 h. The sample was kept and dried at 120 °C for 12 h. The catalyst was calcined for 5 hours with air flowed at 400 °C. Therefore, the process of reduction was conducted for 3 hours with the flow of H₂ gas at a temperature of 450 °C, as reported in previous study [21-23].

2.3 Catalyst characterization

The characterization of catalysts used XRD, BET, and EDX for the Zn-Mo/HZSM-5 catalysts as reported in previous studies [23]. Furthermore, the Zn-Mo/HZSM-5 catalysts has been presented in previous studies [21]. By energy dispersive X-ray, the metals content was 3.25 wt.% of Zinc and 6.34 wt.% of Mo of the total catalyst weight. The sample of the catalyst was denoted as Zn-Mo/HZSM-5_101 catalyst as reported in previous study [23]. Other prepared catalysts with different metal ratios obtained metal content was 2.86 wt.% of Zinc and 5.32 wt.% of Mo of the total catalyst weight from EDX analyzed. The catalyst was named as Zn-Mo/HZSM-5_102.

2.4 Catalytic hydrocracking experiments

Figure 3 shows the equipment used to make biofuel from *Ceiba pentandra* oil by catalytic hydrocracking process using Zn-Mo/HZSM-5 catalyst. The catalytic hydrocracking process was performed using a slurry pressure batch reactor type PARR 4563 with a maximum volume of 600 mL. The reactor was equipped with heating elements. *Ceiba pentandra* oil as much as 200 mL and Zn-Mo/HZSM-5 catalyst as much as 1 g. After the oil and catalyst were prepared in the reactor, the N₂ gas was then flowed into the reactor for 5 minutes to remove oxygen. Then the flow of N₂ was stopped and replaced with H₂ gas for 1 hour to assist the

catalytic hydrocracking reaction process to reach ± 10 -15 bar pressure. The reactor was heated to an operating temperature according to the desired variable and maintained for 2 h. After the catalytic hydrocracking reaction process was over, the reactor was cooled to 30 °C. The reaction temperature was varied to be 300, 325, 350, 375, and 400 °C.

2.5 Product analysis

The liquid hydrocarbon products were analyzed by gas chromatography-mass spectrometry with standard GC: Agilent HP 6890 models 19091S-433, HP-5 MS capillary column 30 m x 250 μm x 0.25 μm . It was held at nominal initial pressure 13.31 psi, initial flow 1.0 mL/min and average velocity 38 cm/sec. The temperature for the oven was at 150 °C for 2 min, then it increased to 240 °C at a rate of 10 °C/min for 13 min. The calculations of biofuel compound was grouped into gasoline, kerosene, and gasoil were based on GC-MS as reported in previous studies [21-23]. The hydrocarbon components with a probability match equal to or higher than 80 % were considered. Hydrocarbons (i.e. paraffin, aromatic, cycloparaffin, and olefin) contained in biofuel were defined as gasoline-like hydrocarbon (C₅-C₉), kerosene-like hydrocarbon (C₁₀-C₁₃), and gasoil-like hydrocarbon (C₁₄-C₂₂), as reported by Barron *et al.* [25].

The liquid hydrocarbon product of *Ceiba*

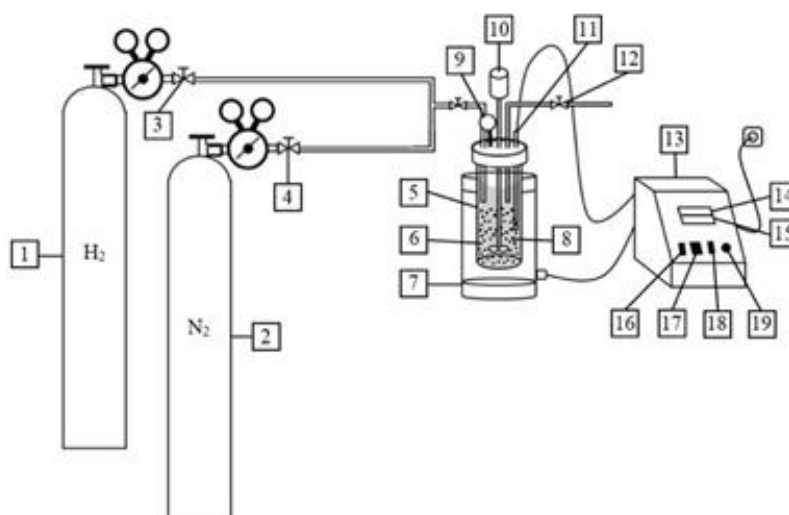


Figure 3. Catalytic hydrocracking process equipment, consisting of: 1) Gas tube H₂, 2) Gas tube N₂, 3) Tube valve gas H₂, 4) Tube valve gas N₂, 5) Reactor, 6) Cotton seed oil (*Ceiba pentandra*), 7) Heater, 8) Catalyst, 9) Reactor pressure indicator, 10) Stirrer, 11) Thermocouple, 12) Control panel pressure batch reactor, 13) Pressure release control, 14) Display temperature of reactor, 15) Display set point temperature, 16) Power control panel heater and stirrer, 17) Power heater button, 18) Stirrer power button and 19) Stirrer speed control keys

pentandra oil were performed by GC-MS and Fourier Transformed Infra Red (FTIR) spectroscopy analyses. Definition of biofuel conversion and selectivity were shown in Equation (1) and (2):

$$\text{Conversion}(\%) = \frac{X_A - X_P}{X_A} \times 100 \quad (1)$$

$$\text{Selectivity}(\%) = \frac{P_P}{P_{HC}} \times 100 \quad (2)$$

where X_A is the % area of carboxyl acid feed, X_P is % area of carboxyl product, P_P is the n-paraffin in product, and P_{HC} is %area total hydrocarbon.

3. Results and Discussion

3.1 Composition of *Ceiba pentandra* oil

The triglyceride fatty acid composition of *Ceiba pentandra* oil are given in Table 1, which shows the triglyceride fatty acid profile of linoleic acid (75.85 %), palmitic acid (21.12 %), and pelargonic acid (0.03 %). The same result was reported by Kathirvelu *et al.* [26], i.e. stated that the largest fatty acid composition in *Ceiba pentandra* seed oil is linoleic acid of 37.45 %. The high content of unsaturated fatty acid makes it a stable liquid at room temperature [26]. The dominant composition of *Ceiba pentandra* oil including a group of unsaturated fatty acids (poly-unsaturated fatty acids, PUFA). Polyunsaturated fatty acids are fatty acids composed of molecules of carbon atoms having more than one double bond. The double bond causes these fatty acid molecules to become unsaturated with hydrogen atoms [27]. Physicochemical analysis was performed on *Ceiba pen-*

tandra oil to analyze the quality of feedstock. The results are showed in Table 2. The acid value is an important parameter to indicate the quality, age and purity degree of an oil [11]. High acid numbers indicate that the free fatty acid content in the oil is high, causing the oil to easily undergo acidification and discoloration. The results of the present study revealed that the acid value of *Ceiba pentandra* oil was 89.49 mg KOH/g. The density (at 20 °C), kinematic viscosity (at 40 °C), and flash point of *Ceiba pentandra* oil were 0.891 g/cm³, 37.47 Cst and 308 °C, respectively.

3.2 Catalyst characterization

Figure 4 (a) shows N₂ adsorption and desorption isotherm as type I, as reported by the previous study [28,29]. The HZSM-5 shows a typical isotherm for microporous materials. After impregnation with Zinc and Mo as shown in Figure 4 (b), the zeolite exhibits a typical irreversible type IV adsorption and desorption isothermal. This characteristic indicates microporous molecular sieves. The pore size distribution indicates that two type pores in the zeolite centered at 3 nm and 10 nm were calculated by the BJH method. It can be seen that the curves of adsorption and desorption are approximately parallel from $P/P_0 = 0.5$ to $P/P_0 = 0.9$.

3.3 Catalytic hydrocracking performance

The FT-IR spectra of *Ceiba pentandra* oil may be compared to that of biofuel obtained at 400 °C for 2 h using Zn-Mo/HZSM-5-102 catalyst to evaluate catalytic hydrocracking reaction such as deoxygenation and decarboxyla-

Table 1. The composition of fatty acid triglyceride of *Ceiba pentandra* oil

Fatty acid triglyceride	Systematic Name	Formula	% Qualitative
Pelargonic acid	Nonanoic acid	C ₉ H ₁₈ O ₂	0.03
Palmitic acid; C16:0	n-Hexadecanoic acid	C ₁₅ H ₃₁ COOH	21.12
Linoleic acid; C18:2	9,12-Octadecadienoic acid	C ₁₇ H ₃₁ COOH	78.85

Table 2. Phycochemical properties of *Ceiba pentandra* oil used in the work

Properties	Value	Method
Density (g/cm ³)	0.891	-
Kinematic viscosity at (40°C, Cst)	37.47	ASTM D445-97
High Heating Value (Btu/lb)	19168	ASTM D240
Acidic Index (mg KOH/g)	89.49	SNI 7431:2008
Iodine value (g.I ₂ /100 g)	52.15	SNI 7381:2008
Flash Point, °C	308	ASTM D93

tion through the disappearance of the absorption peaks corresponding to functional groups of oxygenated compounds as shown in Figure 5. Figure 5a shows FTIR spectra *Ceiba pentandra* oil. The bonding type of some functional groups in *Ceiba pentandra* oil shows the presence of

the bonds of the carboxylic acid group in the absorption area of 3008.06 cm^{-1} , the saturated or unsaturated alkanes in the absorption area 2921.79 cm^{-1} and 2852.56 cm^{-1} and the ester in the absorption area 1724.87 cm^{-1} and 1159.35 cm^{-1} . This result is in accordance with the re-

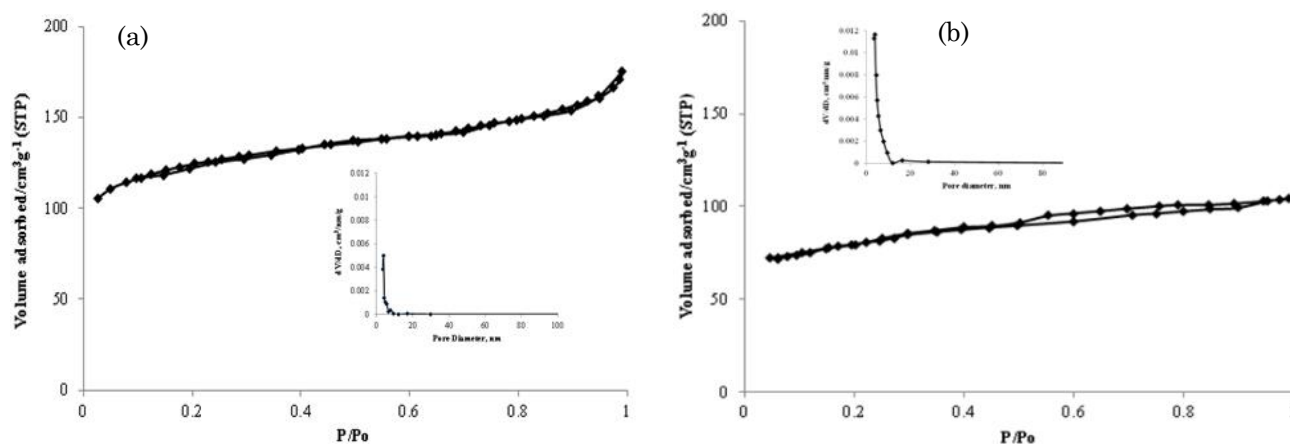


Figure 4. N_2 adsorption and desorption isotherm and pore distribution of (a) HZSM-5, (b) Zn-Mo/HZSM-5_102 catalyst

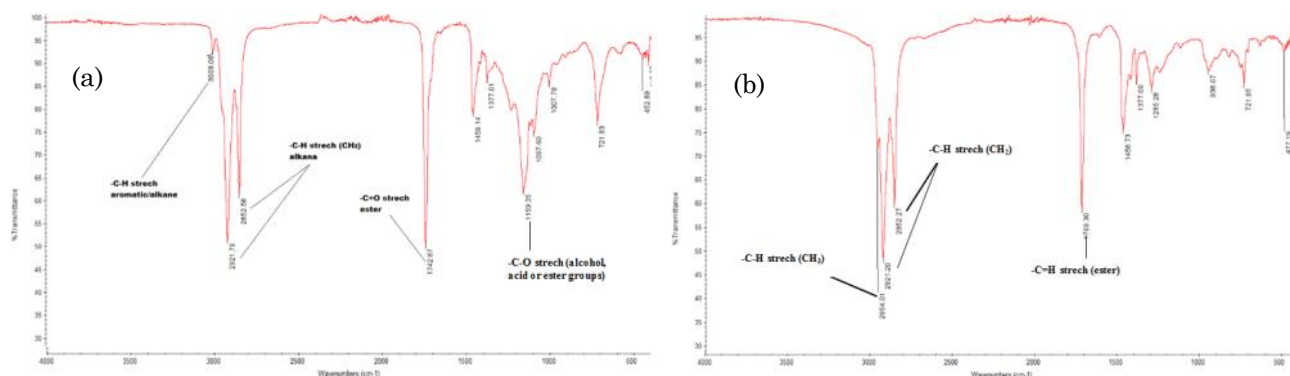


Figure 5. FTIR spectra of (a) *Ceiba pentandra* oil, (b) liquid hydrocarbon product obtained at $400\text{ }^\circ\text{C}$ for 2 h using Zn-Mo/HZSM-5_102 catalyst

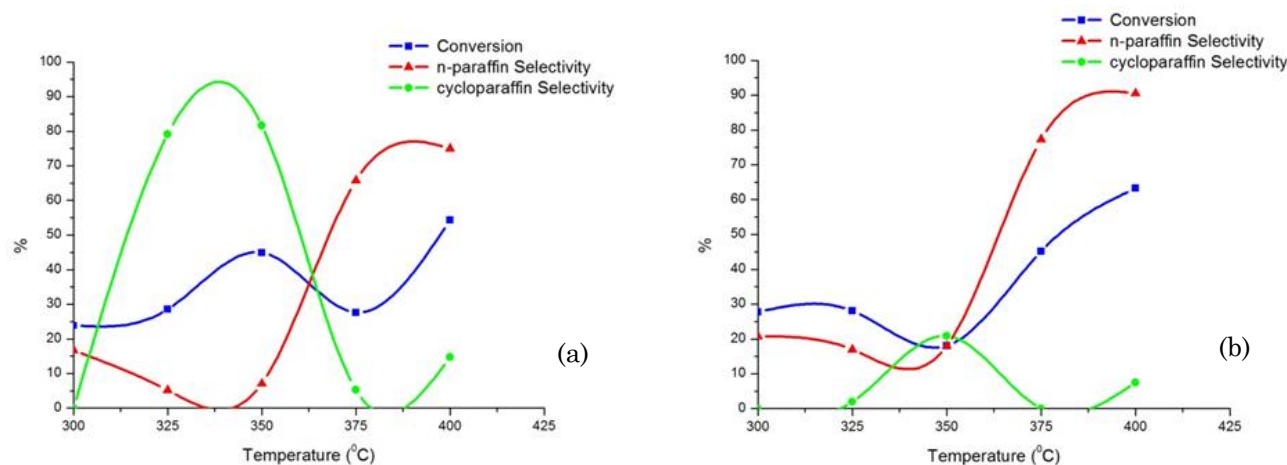


Figure 6. The effect of reaction temperature on conversion and selectivity of liquid hydrocarbon products using Zn-Mo/HZSM-5_102 catalyst type

sults of GC-MS analysis that has been done in the previous study [22].

Figure 5b shows the FTIR spectra of liquid hydrocarbon product at 400 °C with Zn-Mo/HZSM-5_102 catalyst. The spectra showed that the functional group of the carbonyl ester group 1724.87 cm^{-1} and the acid or ester group 1159.35 cm^{-1} were undetectable in liquid hydrocarbon product. This shows that oxygen removal has occurred through the process of catalytic cracking and hydrogenation of ester bonds. The adsorbed hydrocarbons are clearly visible on the region between 2954.01 , 2921.20 , and 2852.27 cm^{-1} for alkyl groups of C–H bonds. This proves that the presence of ZnO and MoO₂ metals are distributed in HZSM-5, as has been previously reported [21], proved capable of hydrogenated the linoleic acid triglycerides *Ceiba pentandra* oil into hydrocarbon compounds. In addition, the catalytic hydrocracking process is affected by the presence of HZSM-5 as support.

Figure 6a shows the effect of the Zn-Mo/HZSM-5_101 catalyst type on the conversion and selectivity of the resulting liquid hydrocarbon product. The highest conversion of 54.35 % was achieved at 400 °C. At 300 °C, the paraffin selectivity reached 16 % and then decreased and increased sharply from 350 to 400 °C. According to the performance of the Zn-Mo/HZSM-5_101 catalyst in the catalytic hydrocracking process on the cotton seed oil, the selectivities of n-paraffin and cycloparaffin were 75.00 % and 14.73 % at temperature of 400 °C, respectively. Figure 6b shows the effect of Zn-Mo/HZSM-5_102 catalyst type on the conversion and selectivity of the resulting liquid hydrocarbon product. The highest conversion of 63.31 % was achieved at temperature of 400 °C. At temperature of 300 °C, the paraffin selectivity reached 20.75 % and then decreased and increased sharply from 375 to 400 °C. According to the performance of the Zn-Mo/HZSM-5_102 catalyst in the catalytic hydrocracking process on the cotton seed oil, the n-paraffin, and cycloparaffin selectivities were 90.52 % and 7.44 % at temperature of 400 °C, respectively.

4. Conclusions

Bifunctional catalytic hydrocracking catalysts, Zn-Mo/HZSM-5, were prepared by incipient wetness impregnation method. The production of *Ceiba pentandra* gasoil range hydrocarbon was used Zn-Mo/HZSM-5 successfully to produce high conversion and selectivity in various reaction temperature. The maximum conversion performance is given by Zn-Mo/HZSM-5_102 catalyst to produce gasoil range hydro-

carbons through catalytic hydrocracking of *Ceiba pentandra* oil of 63.31 %. The n-paraffin selectivity was 90.52 %, while that of cycloparaffin was 7.44 % at 400 °C.

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